

**Low-temperature ashing (LTA) approach for assessing the physically protected organic matter in soil aggregates**

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**Abstract**

Physical stabilization of soil organic matter (SOM) was assessed by a Low-temperature ashing (LTA) approach in cultivated or afforested minesoils with different species for 30 years. Aggregates of 0.5 to 1.0 mm were subjected to LTA by oxygen plasma, a technique able to progressively remove SOM with minimal or no damage to mineral constituents and soil fabric. All investigated minesoils had behaved as C sinks, although to a different extent depending on land use, the cultivated soil storing around half C than the afforested ones. The C in the aggregates after 48h of LTA treatment ranged from 49 to 65% of total C, depending on land use, and it was able to resist to longer treatments. This C was confidently assumed as the one contained in the inner side of these aggregates, and the best protected from decay. The study showed the effectiveness of LTA to distinguish soil C pools benefiting from different physical protections within aggregates and confirmed that this technique could give an important support for assessing the potential of soils to sequester C and/or of the responses of individual ecosystems to changes in land use and management. In addition, this approach could be useful for the establishment of the baseline of organic C level in different soils. In fact, it could give the actual amount of stabilized C in the soil.

*Keywords: Low-temperature ashing (LTA); carbon sequestration; physical protection; soil structure; minesoils*

**Introduction, scope and main objectives**

The assessment of stabilised OM in soil aggregates is of paramount importance for implementing strategies to increase C sequestration in soil and consequently mitigate climate change.

Soil organic carbon dynamics are driven not only by the intrinsic properties of the organic matter itself but also by the environmental and biological influences, which may reduce the rate of decomposition, thereby allowing the organic matter to persist for long time (Schmidt et al., 2011). The physical protection of C in soil aggregates is a sound parameter to describe the processes that affect directly or indirectly the sequestration of C in soil. The main objective of this work is revealing the possibility to measure the actual C stabilized in soil aggregates. For this purpose a reclaimed minesoil, cultivated or afforested with different species since 30 years was investigated. In particular, the studied land uses were: 1) a managed (thinned and mowed) English oak (*Quercus robur* L.) plantation; 2) a similarly managed 1:1 mixed plantation of Italian alder (*Alnus cordata* Loisel.) and English oak; 3) an unmanaged portion of the mixed plantation; 4) a cropland tilled and manured every year (D'Acqui et al., 2017).

**Methodology**

The proposed approach to disentangle the role of physical protection of aggregates to SOM is based on Low-Temperature Ashing (LTA) by oxygen plasma, which enables a controlled removal of SOM from the surface of soil samples inwards without damaging the inorganic constituents or the aggregate fabric. On this basis, it is possible to obtain a dynamic of C removal from soil aggregates. The LTA treatment was

performed by the self-assembled equipment described in D'Acqui et al. (1999). Aggregates of 0.5 to 1.0 mm were allocated in the LTA reactor and evacuated to 45 Pa under an oxygen 20 mL min<sup>-1</sup> flow rate. Oxygen plasma was produced applying a radiofrequency of 13.56 MHz by a power input of 100 W and a reflected power of 5 W. In these conditions the surface temperature was maintained below 80 °C. Carbon was measured by dry combustion with a Carlo Erba NA 1500 CHNS Analyzer (Milan, Italy) on three aliquots (~1 g) per sample exposed to different treatment times, i.e. 5h, 24h, and 48h by LTA. No further C removal was noticed beyond 48h, which was therefore selected as longest LTA exposition time and the residual C assumed as “physically protected C. Such physical protection was however proved by the fact that, after grinding the 48h LTA treated aggregates and successively subjected again to LTA, they lost almost completely their C content.

## Results

The C removal from the aggregates is related to the nature and organization of soil particles that, in turn, determine the size, shape and network of pores and the exposition of organic matter at the plasma-substrate interface. The diffusion of plasma into microaggregates is low, similarly to gases in soil, hence its oxidative power mimics natural oxidative processes. There is a relatively rapid reduction of C in the first 5 hours of treatment (Figure 1), then the slope decreases much up to reach a “plateau” phase at around 20 h, when evidently no further C is removed.

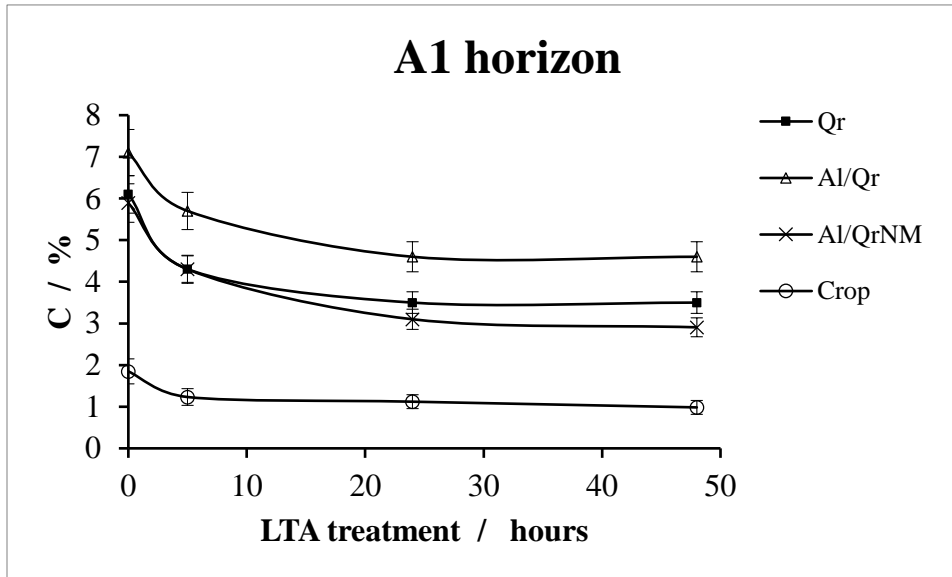


Fig. 1: C removal by LTA from 0.5-1.0 mm aggregates in the A1 horizon of afforested or 0-5 cm layer in cropped soil (D'Acqui et al., 2017). Qr=managed English oak (*Quercus robur* L.) plantation, Al/Qr=managed mixed plantation of English oak and Italian alder (*Alnus cordata* Loisel.), Al/QrNM=unmanaged mixed plantation of English oak and Italian alder, Crop= cropland.

**Table 1: Residual C in 0.5-1.0 mm aggregates after different times of LTA treatment. Total C is the one measured in untreated aggregates, i.e. 0 h LTA treatment. The residual C after 48h of LTA treatment was assumed as “protected C”. Number in brackets are standard deviations of n=3. (D’Acqui et al., 2017)**  
**Qr=managed English oak (*Quercus robur* L.) plantation, Al/Qr=managed mixed plantation of English oak and Italian alder (*Alnus cordata* Loisel.), Al/QrNM=unmanaged mixed plantation of English oak and Italian alder, Crop=cropland..**

| <i>Residual C</i> |              |              |                |             |
|-------------------|--------------|--------------|----------------|-------------|
| <b>A1 horizon</b> |              |              |                |             |
|                   | <b>Qr</b>    | <b>Al/Qr</b> | <b>Al/QrNM</b> | <b>Crop</b> |
| LTA treatment     | <b>C (%)</b> |              |                |             |
| 0 h               | 6.1 (±0.3)   | 7.1 (±0.3)   | 5.9 (±0.3)     | 1.8 (±0.2)  |
| 5 h               | 4.3 (±0.2)   | 5.7 (±0.3)   | 4.3 (±0.2)     | 1.2 (±0.1)  |
| 24 h              | 3.5 (±0.2)   | 4.6 (±0.2)   | 3.1 (±0.1)     | 1.1 (±0.1)  |
| 48 h              | 3.5 (±0.2)   | 4.6 (±0.2)   | 2.9 (±0.1)     | 1.0 (±0.1)  |

## Discussion

Figure 1 and Table 1 both show that almost half of initial C content (0h LTA treatment) of all soils was physically protected. Protection within the aggregates entails the inaccessibility of soil microbes to organic compounds and a limitation in O<sub>2</sub> availability with the consequent reduction of bio-chemical activities (Six et al., 2004; von Lütow et al., 2006; Stockmann et al., 2013). As a consequence, the physically protected SOM undergoes lower rate of decomposition and longer turnover time than the rest of SOM (Shrestha and Lal, 2006; Stockmann et al., 2013). The significant C loss in the first 5 hours of LTA treatment (around 30%) both in the forest and the cropland is most probably due to the oxidation of matter located in easily accessible niches of aggregates and not closely associated with minerals (Stockmann et al., 2013). Such a SOC fraction could be assumed as the one most prone to bio-chemical decomposition processes, hence with shorter turnover time. On the other hand, the SOC fraction oxidised by further LTA treatment, i.e. the one removed between 5h and 48h of treatment, which amounts to around the 22% of initial C in afforested soils and 6% in the cultivated soil, respectively, can be considered as partially protected, because unreachable or intimately bound to mineral particles. Such organic fraction can be assumed to have intermediate turnover time.

## Conclusions

The approach used in this study provided insights into the amount of “physically protected C” in minesoils and confirmed that the LTA technique could give an important help for the assessment of the potential of soils in sequestering C and/or of the responses of individual ecosystems to changes in land use and management. In addition, this approach could be useful for the establishment of the baseline of organic C level in different soils. In fact, it could give the actual amount of stabilized C in the soil.

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